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THE REACTION OF DISSOLVED SILVER WITH VARIOUS SULPHIDE MINERALS IN SULPHATE AND CHLORIDE MEDIA

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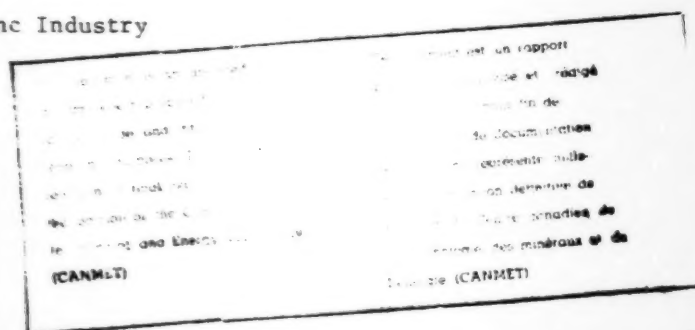
September 1984

Project: MRP-3.4.0.3.10
Silver Recovery in the Zinc Industry

33 pp

MINERALS RESEARCH PROGRAM
MINERAL SCIENCES LABORATORIES
DIVISION REPORT MRP/MSL 84-128 (TR)

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THE REACTION OF DISSOLVED SILVER WITH
VARIOUS SULPHIDE MINERALS IN SULPHATE AND CHLORIDE MEDIA

by

D.J. MacKinnon* and J.M. Brannen**

ABSTRACT

The reaction of dissolved silver with various sulphide minerals has been studied in both sulphate and chloride media. The effects of temperature, stirring rate, mineral particle size, initial silver concentration, ferric ion concentration and ferrous ion concentration on the extent of reaction between dissolved silver and various sulphide minerals were determined.

In sulphate media, the order of reactivity of dissolved silver with sulphide minerals was:

chalcocite ~ covellite > bornite > pyrite >> galena > sphalerite >
chalcopyrite

Increasing temperature and stirring rate had only a slight effect on the reaction. The percentage Ag reacted increased linearly with increasing amounts of the mineral sulphide and with decreasing mineral particle size. The rate of reaction of dissolved Ag with chalcopyrite increased linearly with increasing initial Ag concentration. With the exception of galena, the percentage Ag reacted with sulphide minerals decreased when Fe^{+3} was added to the solution.

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In chloride media only galena and sphalerite reacted with dissolved Ag to a significant extent. The addition of Fe^{+3} also decreased the percentage Ag reacted with these minerals.

RÉACTION DE L'ARGENT DISSOUS AVEC DIVERS MINÉRAUX
SULFURÉS DANS DES MILIEUX SULFATÉS ET CHLORURÉS

par

D.J. MacKinnon* et J.M. Brannen**

RÉSUMÉ

On a étudié à la fois dans les milieux sulfatés et chlorurés la réaction de l'argent dissous avec divers minéraux sulfurés. On a déterminé les effets de la température, de la vitesse d'agitation, du calibre des minéraux, de la concentration initiale d'argent et de la concentration des ions ferriques et ferreux sur l'importance de la réaction entre l'argent dissous et divers minéraux sulfurés.

Dans les milieux sulfatés, l'ordre de réactivité de l'argent dissous vis-à-vis des divers minéraux sulfurés était le suivant:

chalcocite ~ covellite > bornite > pyrite » galène >
sphalérite > chalcopyrite.

L'augmentation de la température et de la vitesse d'agitation n'avaient qu'un effet léger sur la réaction. Le pourcentage d'Ag réagissant avec les minéraux sulfurés augmentait linéairement avec l'augmentation des quantités de minéraux sulfurés et avec la diminution de la granulométrie. La vitesse de réaction d'Ag dissous en présence de la chalcopyrite augmentait linéairement avec l'augmentation de la concentration initiale d'Ag. Sauf en présence de la galène, le pourcentage d'Ag réagissant avec

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les minéraux sulfurés diminuait lorsqu'on ajoutait Fe^{+3} à la solution.

Dans les milieux chlorurés, seules la galène et la sphalérite réagissaient avec l'argent dissous dans des proportions significatives. L'ajout de Fe^{+3} a aussi diminué le pourcentage d'Ag réagissant avec ces minéraux.

INTRODUCTION

The purpose of this investigation was to determine the behaviour of silver in the presence of sulphide minerals in both sulphate and chloride media with and without ferric/ferrous ions. Such reactions are important, of course, in the ferric ion leaching of sulphide minerals, but are equally important in zinc processing circuits where soluble silver could react in the roaster product (1). Silver losses occur in a manner that is not completely understood. A possible loss of silver occurs in the leaching operation where dissolved silver may precipitate (e.g., as Ag_2S) on the surface of residual sulphide mineral particles. Thus the present study was undertaken to determine the extent to which dissolved silver reacted with various sulphide minerals and also to determine the relative reactivity of the various sulphide minerals with dissolved silver.

Previously, Kamenetski (2) reported that Ag_2S precipitated and sulphide cations dissolved when small plates of chalcopryrite, pyrite, galena, covellite and sphalerite were contacted with 0.01 N AgNO_3 solution. He also reported (3) that the rate of reaction of silver nitrate was proportional to the mineral surface area and that the rate had a characteristic value for each sulphide.

More recently, Miller et al. (4) reported that Ag^+ additions had a catalytic effect on the ferric sulphate leaching of chalcopryrite. The enhanced leaching rate was attributed to the formation of an intermediate Ag_2S film on the chalcopryrite surface by an exchange reaction.

Perrotel et al. (5) studied the behaviour of silver ions in the presence of sphalerite. They found that silver reacted rapidly with sphalerite and SEM observations indicated the formation of a polycrystalline layer of acanthite, Ag_2S , epitactically oriented on the zinc sulphide surface.

EXPERIMENTAL

The reaction of dissolved silver with sulphide minerals was carried out in a vessel containing 300 mL of the desired solution. The vessel was fitted with a cover designed to hold and position a thermometer, nitrogen gas sparger and a stirring shaft; the cover also contained a sampling port from which solution was withdrawn at timed intervals for analysis by atomic absorption spectrophotometry. The reaction vessel was placed in a thermostated water bath to maintain the temperature, generally at 70°C.

The sulphate solution consisted of 0.3 M H_2SO_4 . The desired concentrations of silver, ferric and/or ferrous ion were obtained by adding the appropriate amount of the respective sulphate salts.

The chloride solution consisted of 0.3 M HCl and 3 M NaCl (required to solubilize the AgCl). The desired concentrations of silver, ferric and/or ferrous ion were obtained by adding the appropriate amount of the respective chloride salts.

A variety of granulated mineral sulphides including: chalcopyrite, pyrite, galena, sphalerite, chalcocite, covellite and bornite were used in the tests. A known weight, usually 0.5 g, of the sized mineral was added to the solution to initiate the reaction. A specially designed plexiglass stirrer, Figure 1, was used to provide intimate mixing of the mineral particles and solution. The stirrer, 2.0 inches in diameter and $\frac{1}{4}$ -inch thick, was connected to a $\frac{1}{4}$ -inch diameter Plexiglas shaft. Four equally spaced $\frac{3}{8}$ -inch holes were drilled at 60° angles to the central shaft around the flat surface of the disk and $\frac{1}{4}$ -inch from its edge. Solution samples were withdrawn through a glass frit to ensure that no mineral particles were entrained.

RESULTS AND DISCUSSION

SULPHATE MEDIAChalcopyrite

The reaction of dissolved silver with chalcopyrite was studied using 0.5 g of the sized mineral in a solution containing 0.3 M H_2SO_4 and 60 mg/L Ag. The solution was maintained at 70°C and stirring at a rate of 1250 RPM. The results of 1-h tests using -200 +325 (-70 +43 μm) and -100 +200 (-140 +70 μm) size fractions of chalcopyrite are presented in Figure 2 as plots of Ag reacted (%) vs time.

As the data in Figure 2 indicate, the particle size has a significant effect on the quantity of Ag removed from solution. Both the rate of Ag removal and the total amount of Ag reacted increased with the finer particle size. For the (-100 +200) size fraction, the reaction virtually stopped after the first few minutes with the result being that only ~20% of the Ag present in solution reacted with the chalcopyrite. For the (-200 +325) size fraction, the rate of Ag removal from solution increased rapidly for about the first 10 minutes and then gradually tapered off; after 60 minutes >90% of the dissolved Ag had reacted with the chalcopyrite.

Reactivity of Metal Sulphides

The results obtained from the reaction of dissolved Ag with different size fractions of various sulphide minerals are summarized in Table 1. In terms of percentage Ag reacted, the order of reactivity for the (-100 +200) size fraction of the various sulphide minerals is as follows:

chalcocite ~ covellite > bornite > pyrite >> galena > sphalerite >
chalcopyrite

Table 1. Effect of Particle Size on Percentage Silver Reacted with Various Sulphide Minerals

Mineral (0.5 g)	Ag Reacted (%)		
	(-100 +200)	(-200 +325)	(-280 +370)
	(-140 +70 μm)	(-70 +43 μm)	(-49 +37 μm)
Chalcocite	99	99	-
Covellite	100	-	-
Bornite	95	-	-
Pyrite	88	88	-
Galena	45	97	-
Sphalerite	31	90	-
Chalcopyrite	19	91	99

0.3 M H_2SO_4 ; 70°C; 1250 RPM; 1 h; $[\text{Ag}]_i = 60 \text{ mg/L}$

For the (-200 +325) particle size fraction, all minerals studied showed the same high reactivity (>90%) towards dissolved Ag. The finer size fractions of galena, sphalerite and chalcopyrite all showed a substantially increased reactivity towards dissolved Ag with respect to the corresponding -100 +200 mesh material.

Effect of Temperature

The effect of temperature on the reaction of dissolved Ag with various sulphide minerals was studied over the range 30-80°C in 0.3 M H_2SO_4 containing 60 mg/L Ag. The data obtained for chalcopyrite, sphalerite and chalcocite are summarized in Table 2. Variations in temperature had a modest effect on the initial rate of reaction of dissolved Ag with these sulphide minerals. Increasing the temperature from 60 to 70°C resulted in an increase in the percentage Ag reacted with sphalerite and chalcopyrite. With chalcocite, 99% of the Ag reacted even at 30°C. At 50°C, the percentage of Ag removed from solution increased in the order chalcocite > chalcopyrite > sphalerite.

Effect of Stirring Rate

The effect of stirring rate on the percentage Ag reacted with sulphide minerals was studied over the range 750-1250 rpm using 0.3 M H_2SO_4 with 60 mg/L Ag. The data obtained for chalcopyrite and sphalerite are summarized in Table 3. Increasing the stirring rate had no significant effect on the initial rate of reaction of dissolved silver with these minerals. There was, however, some variation in the percentage Ag reacted with stirring rate as indicated in Table 3.

Table 2. Effect of Temperature on the Reaction of Dissolved Silver with Various Sulphide Minerals

Mineral (0.5 g)	Ag Reacted				
	30°C	50°C	60°C	70°C	80°C
Chalcopyrite (-280 +370)	-	81	89	99	97
Sphalerite (-200 +325)	-	53	52	84	-
Chalcocite (-100 +150)	99	98	-	99	-

0.3 M H_2SO_4 ; 1250 RPM; 1 h; $[\text{Ag}]_i = 60 \text{ mg/L}$

Table 3. The Effect of Stirring Speed on the Reaction of Dissolved Silver with Various Sulphide Minerals

Mineral (0.5 g)	Ag Reacted (%)		
	750 rpm	1000 rpm	1250 rpm
Chalcopyrite (-280 +370)	88	87	99
Sphalerite (-200 +325)	74	64	84

0.3 M H_2SO_4 ; 70°C ; 1 h; $[\text{Ag}]_i = 60 \text{ mg/L}$

Effect of the Initial Ag Concentration

The effect of the initial silver ion concentration on the reaction of dissolved Ag with 0.5 g of chalcopyrite (-280 +370) was studied over the range 15-90 mg Ag/L in 0.3 M H_2SO_4 solution at 77°C. The results are presented in Figure 3 in terms of the total Ag reacted (in mg) over a one hour period as a function of the initial silver concentration (mg/L). As shown in Figure 3, the amount of Ag reacted is directly proportional to the initial Ag concentration in solution.

Effect of the Amount of Sulphide Mineral

The effect of the amount (weight) of sulphide mineral on the rate of reaction of dissolved Ag was studied over the range 0.5 to 1.5 g using (-280 +370) chalcopyrite in 0.3 M H_2SO_4 containing 60 mg/L Ag at 70°C. The results are presented in Figure 4 as a plot of initial reaction rate (mg/min) vs weight of chalcopyrite (g). As indicated in Figure 4, the initial rate of Ag reacted is directly proportional to the amount of chalcopyrite added to the solution. Hence, the rate is directly proportional to the area of the sulphide presented to the dissolved silver.

The Effect of Ferric Ion Concentration

The presence of ferric ion in the solution resulted in a significant decrease in the percentage Ag reacted with chalcopyrite. Figure 5 shows the percentage silver reacted vs time plots for solutions containing 0, 0.1 and 0.3 M Fe^{3+} . In the presence of 0.1 M Fe^{3+} , approximately 25% Ag reacts in the first few minutes. Thereafter, there is only a slight increase in the percentage Ag reacted over the remaining 60 minutes. At 0.3 M Fe^{3+} , the percentage Ag reacted reached a maximum of ~23% after about 7 minutes; the percentage Ag reacted decreased over the remainder of the reaction period indicating a re-solution of Ag.

Figure 6 shows a plot of percentage Ag reacted vs Fe^{+3} concentration in the presence and absence of 0.3 M Fe^{2+} . Increasing the Fe^{+3} concentration in the absence of Fe^{2+} results in a rapid decrease in the percentage Ag reacted to about 0.3 M and thereafter there is a more gradual decrease in the percentage Ag reacted. In the presence of 0.3 M Fe^{2+} , there is a rapid decrease in the percentage Ag reacted to 0.1 M Fe^{3+} ; the decrease is less rapid for $\text{Fe}^{+3} > 0.1 \text{ M}$. At 1.0 M Fe^{+3} , the percentage Ag reacted was only 10% in both the presence and absence of 0.3 M Fe^{2+} . For Fe^{3+} concentrations $< 1 \text{ M}$ the percentage Ag reacted was consistently higher when the solution also contained 0.3 M Fe^{2+} .

The effect of the Fe^{+3} concentration on the percentage Ag reacted with various sulphide minerals is summarized in Table 4. With the exception of galena, there is a significant decrease in the percentage Ag reacted with increasing Fe^{+3} concentration. The most pronounced effect of Fe^{+3} concentration was observed for pyrite where no Ag reacted when the solution contained 0.1 M Fe^{+3} . The effect of Fe^{+3} concentrations $< 0.1 \text{ M}$ on the percentage Ag reacted with pyrite and covellite is shown in Table 5. Thus, the presence of Fe^{+3} is most effective in retarding the reaction of Ag with pyrite, followed closely by covellite and then by chalcopyrite. Fairly concentrated solutions are needed to suppress the reaction with sphalerite and galena.

The results suggest that Ag precipitation would likely occur in the neutral leach circuit of a zinc operation where the Fe^{3+} concentration is $< 1 \text{ g/L}$. In a ferric sulphate leaching process for sulphide concentrates, Ag would likely dissolve early in the leach when the Fe^{3+} concentration was high and then reprecipitate at the end of the leach when the ferric content was exhausted.

Table 4. Effect of Ferric Ion Concentration on the Percentage Silver Reacted with Various Sulphide Minerals (-200 +325 mesh)

Mineral (0.5 g)	Ag Reacted (%)			
	0 M Fe ³⁺	0.1 M Fe ³⁺	0.3 M Fe ³⁺	1.0 M Fe ³⁺
Chalcocite	99	85	62	16
Galena	97	83	97	91
Sphalerite	90	83	70	15
Chalcopyrite	91	52	11	-
Pyrite	88	0	-	-

0.3 M H₂SO₄; 70°C; 1250 RPM; 1-h; [Ag]_i = 60 mg/L

Table 5. Effect of Ferric Ion Concentration on the Percentage Silver Reacted with Pyrite and Covellite

Mineral (0.5 g)	0	Ag Reacted (%)						
		0.001	0.005	0.01	0.02	0.03	0.05	0.10
Pyrite (-200 +325)	88	35	9	5	4	-	-	0
Covellite (-100 +200)	100	-	-	94	-	31	8	6

0.3 M H_2SO_4 ; 70°C; 1250 RPM; 1-h; $[\text{Ag}]_i = 60 \text{ mg/L}$

Reaction Product Examination

The reaction products were examined by scanning electron microscopy (SEM) and the presence of Ag was confirmed using energy dispersive X-ray analysis (EDX). The SEM photomicrographs in Figure 7 compare the residues obtained for the reaction of dissolved Ag with chalcocite in the absence (Fig. 7(a)) and presence (Fig. 7(b)) of 0.3 M Fe^{+3} . In the absence of Fe^{+3} (Fig. 7(a)), an appreciable deposit exists on most grains and EDX showed the Ag content of the deposit to be quite variable.

In the presence of 0.3 M Fe^{+3} (Fig. 7(b)), the surface of the chalcocite particles was entirely different and the grains show evidence of ferric ion leaching. The Ag content was still variable but less so than the variation observed in the absence of Fe^{+3} . The SEM photomicrographs shown in Figure 7(c) and 7(d) compare the residues obtained for the reaction of dissolved Ag with galena in the absence (Fig. 7(c)) and presence (Fig. 7(d)) of Fe^{+3} . With respect to its reactivity with dissolved Ag, galena was least affected by the presence of Fe^{+3} and both residues, Fig. 7(c) and (d) had appreciable Ag on the surface.

CHLORIDE MEDIA

Effect of Particle Size

With the exception of galena and sphalerite, the sulphide minerals did not react with dissolved silver to a significant extent in chloride media. The results obtained for the -200 +325 and -100 +200 size fractions of various sulphide minerals are summarized in Figures 8 and 9.

Figure 8 shows the plots of the percentage Ag reacted vs time obtained for 0.5 g of -200 +325 mesh chalcopyrite, covellite, galena and sphalerite in 3.0 M NaCl , 0.3 M HCl solution containing 30 mg/L Ag at 70°C stirred at

a rate of 1250 RPM. As indicated by the curves in Figure 8, the maximum extent of Ag reaction occurred within the first 5 minutes. For both covellite and chalcopyrite, <10% Ag reacted whereas for galena and sphalerite the maximum amount of Ag reacted was 80 and 85%, respectively. In all cases but particularly for galena, the percentage Ag reacted decreased with increasing reaction time, in fact after 60 minutes, the Ag which had precipitated was completely redissolved.

Similar trends were observed for the -100 +200 size fractions of galena, chalcocite, covellite and sphalerite, Figure 9. For chalcocite, the percentage Ag reacted peaked at ~27% after ~8 minutes, then decreased rapidly to zero after 15 minutes. The percentage Ag reacted with sphalerite was maximized at 30% after 15 minutes, an extent of precipitation much less than that observed for the -200 +325 size fraction of this mineral.

The rate of reaction of dissolved Ag with the -100 +200 size fraction of galena was very rapid, reaching a maximum of ~95% after 5 minutes. The rate of Ag redissolution was less than that observed with the -200 +325 size fraction of galena.

Effect of Ferric Ion Concentration

The effect of ferric ion concentration on the reaction of dissolved Ag with 0.5 g of -100 +200 mesh galena is shown in Figure 10. With the addition of 0.001 M Fe^{+3} to the solution, the rate of reaction of dissolved Ag with galena decreased considerably. The maximum Ag reacted (~87%) occurred at about 35 minutes, after which the Ag began to redissolve. At 0.01 M Fe^{+3} , <5% Ag reacted with the galena. It is known that galena is rapidly leached even in 0.01 M FeCl_3 media and that the surface is easily passivated with PbCl_2 (6).

The presence of 0.001 M Fe^{+3} also reduced the percentage Ag reacted with sphalerite as shown in Figure 11. The maximum Ag reacted, 25%, was achieved after the first 10 minutes and this remained constant for the duration of the test. In the presence of 0.01 M Fe^{+3} , only about 5% Ag reacted with the sphalerite and this amount decreased to zero after about 40 minutes.

CONCLUSIONS

The reaction of dissolved Ag with various sulphide minerals has been studied in both sulphate and chloride media both in the presence and absence of ferric ion. In sulphate media, the order of reactivity of dissolved Ag with sulphide minerals was:

chalcocite ~ covellite > bornite > pyrite >> galena > sphalerite >
chalcopyrite

Increasing temperature and stirring speed had only a slight effect on the reaction. The percentage Ag reacted increased linearly with increasing weights of the sulphide and with decreasing particle size suggesting the reaction was directly proportional to the surface area of the sulphide. The rate of Ag reaction increased linearly with increasing initial Ag concentrations. With the exception of galena, the percentage Ag precipitated onto various sulphide minerals decreased when Fe^{3+} was added to the solution.

In chloride media, only galena and sphalerite reacted with dissolved Ag to a significant extent. The addition of Fe^{+3} also decreased the percentage Ag reacted with these minerals and the effect was appreciable. For Fe^{+3} concentrations $>0.01 \text{ M}$, dissolved Ag did not react with these minerals in chloride media.

ACKNOWLEDGEMENT

Thanks are due to D. Owens for providing the SEM photomicrographs.

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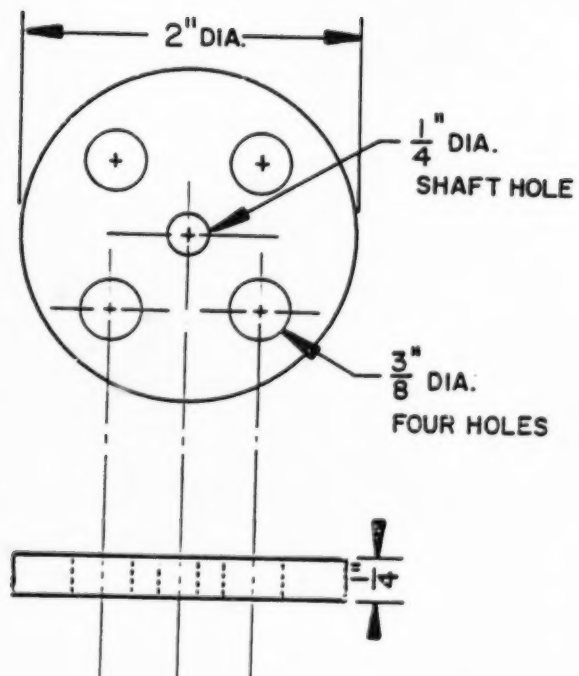


FIG. 1

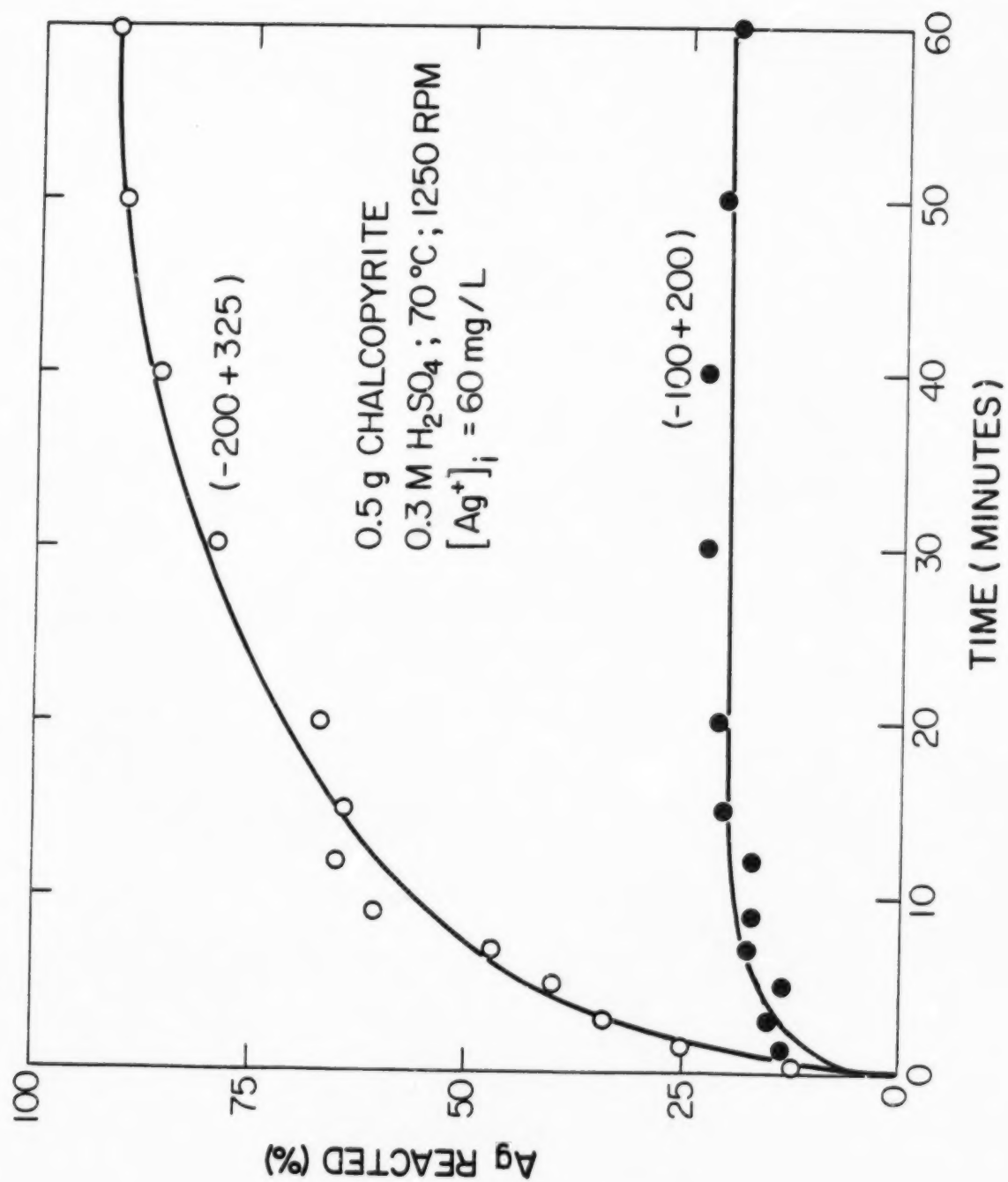


FIG. 2

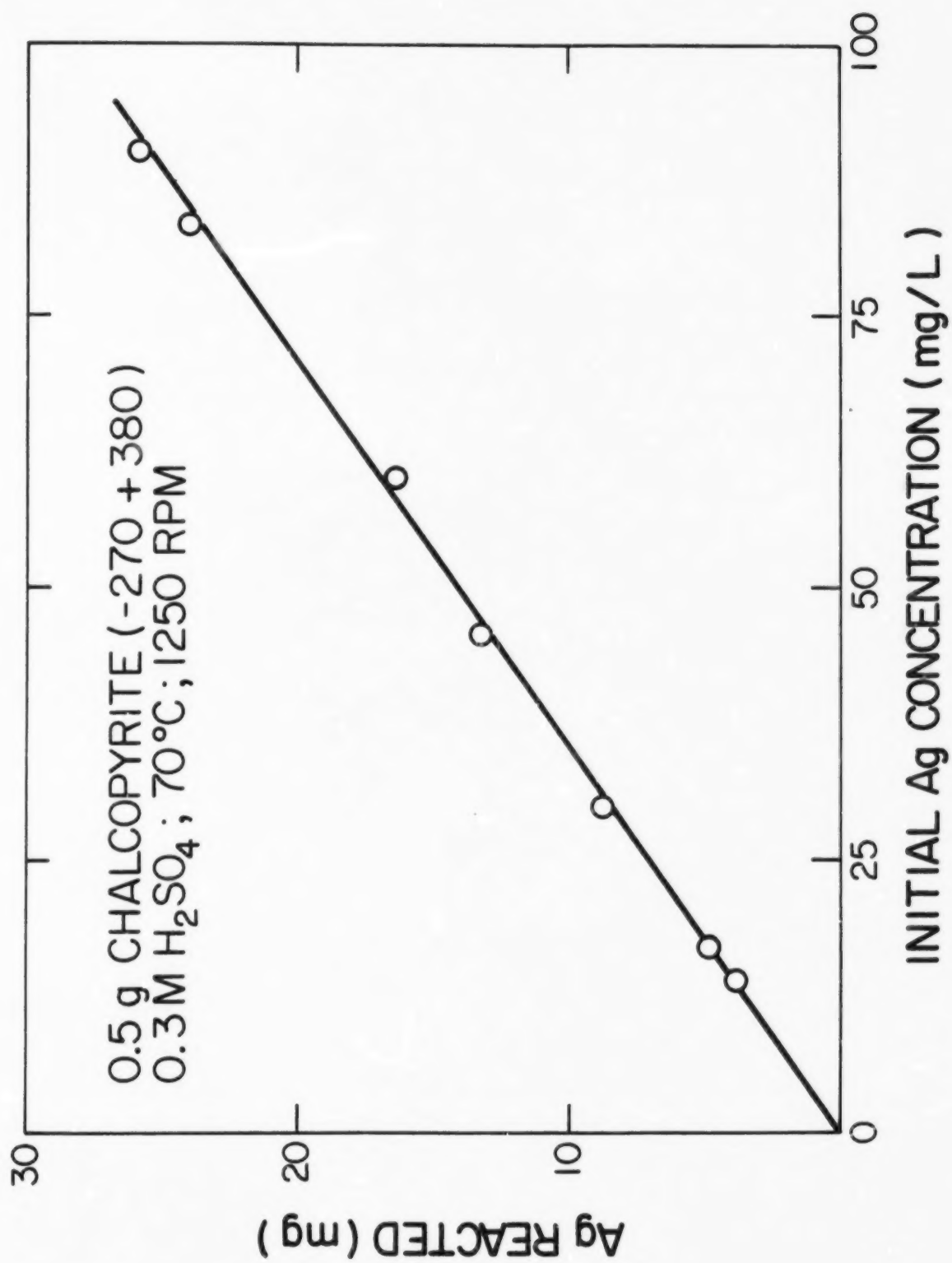


FIG. 3

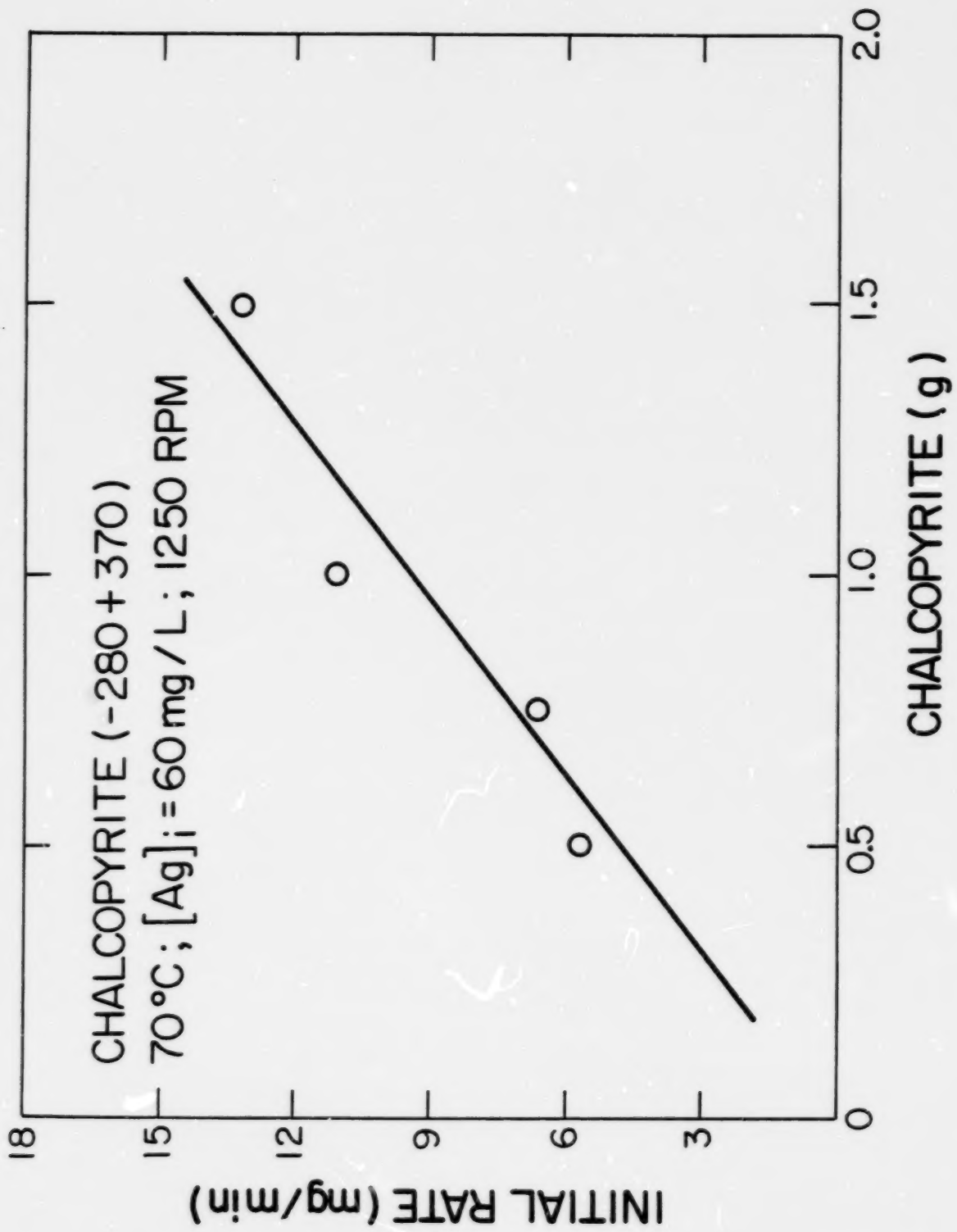


FIG. 4.

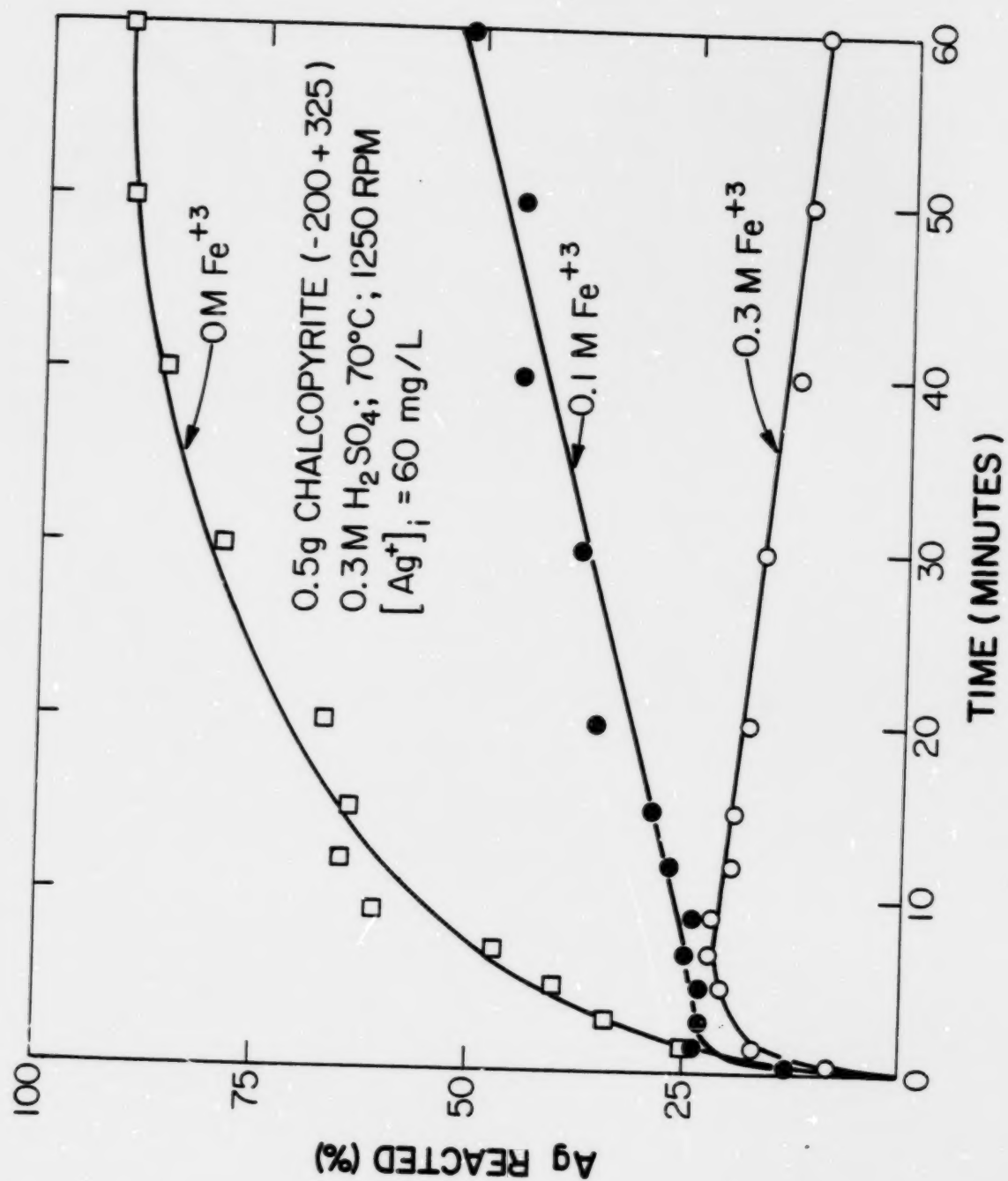


FIG. 5

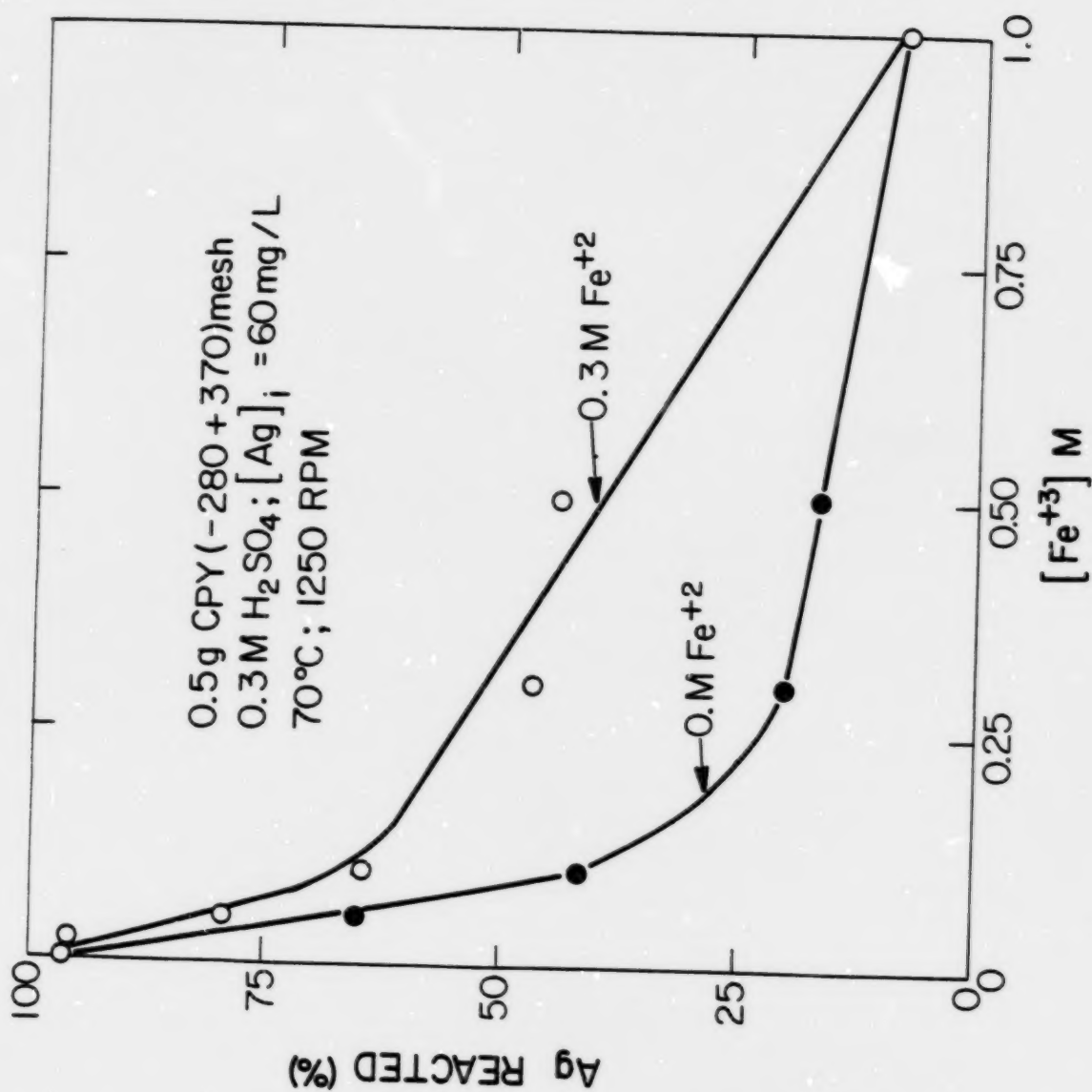
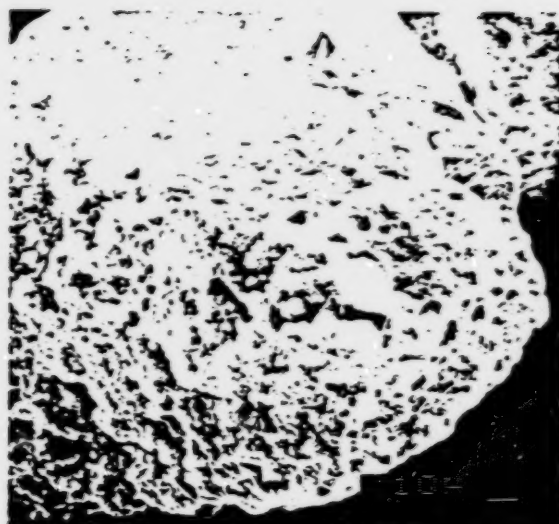


FIG. 6



a) Chalcocite (-100 +150 mesh)
No Fe^{+3}



b) Chalcocite (-100 +150 mesh)
0.3 M Fe^{+3}



c) Galena (-200 +325 mesh)
No Fe^{+3}



d) Galena (-200 +325 mesh)
0.3 M Fe^{+3}

Figure 7. SEM photomicrographs of residues obtained from the reaction of dissolved Ag with chalcocite and galena in the presence and absence of ferric iron.

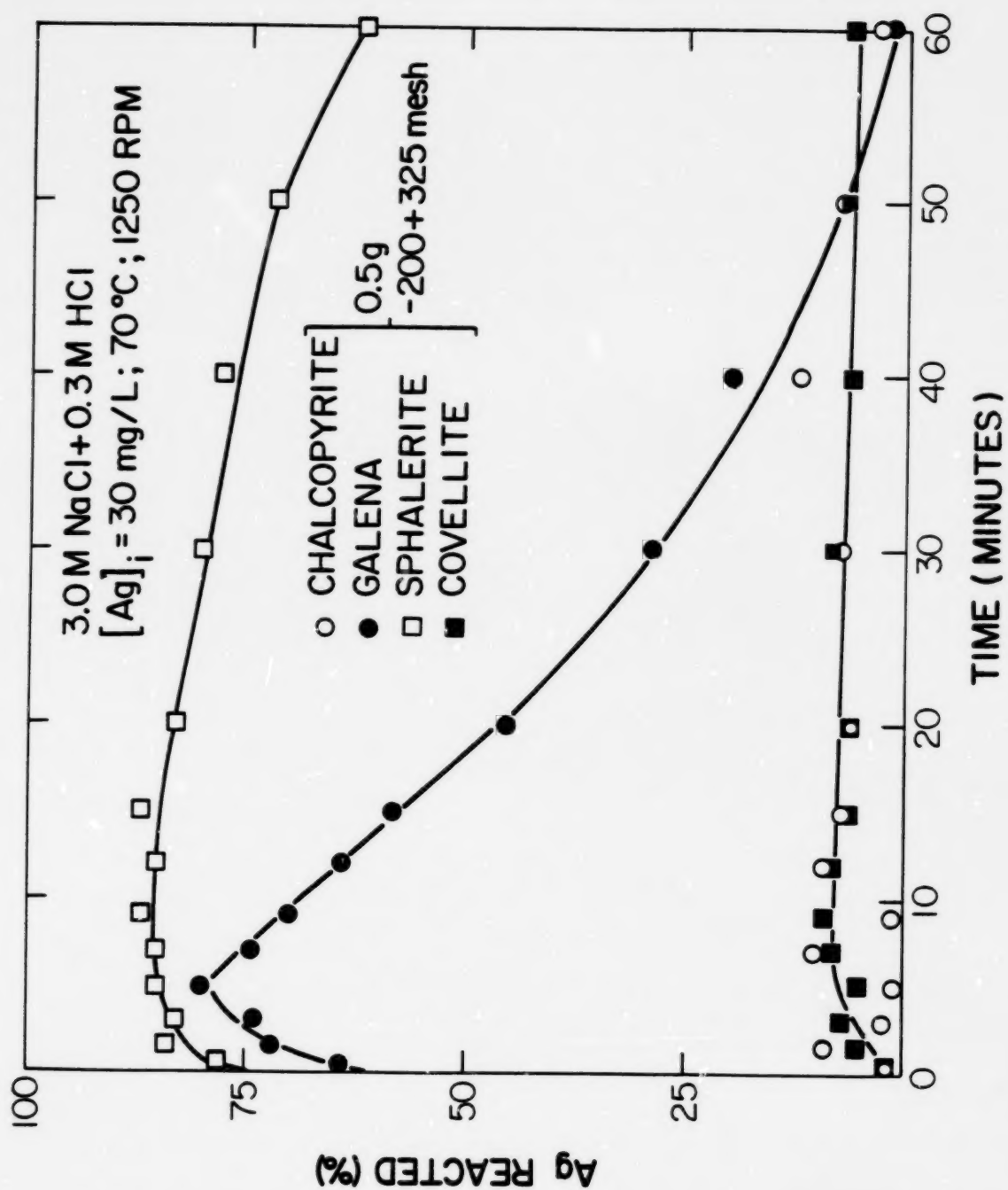


FIG. 8

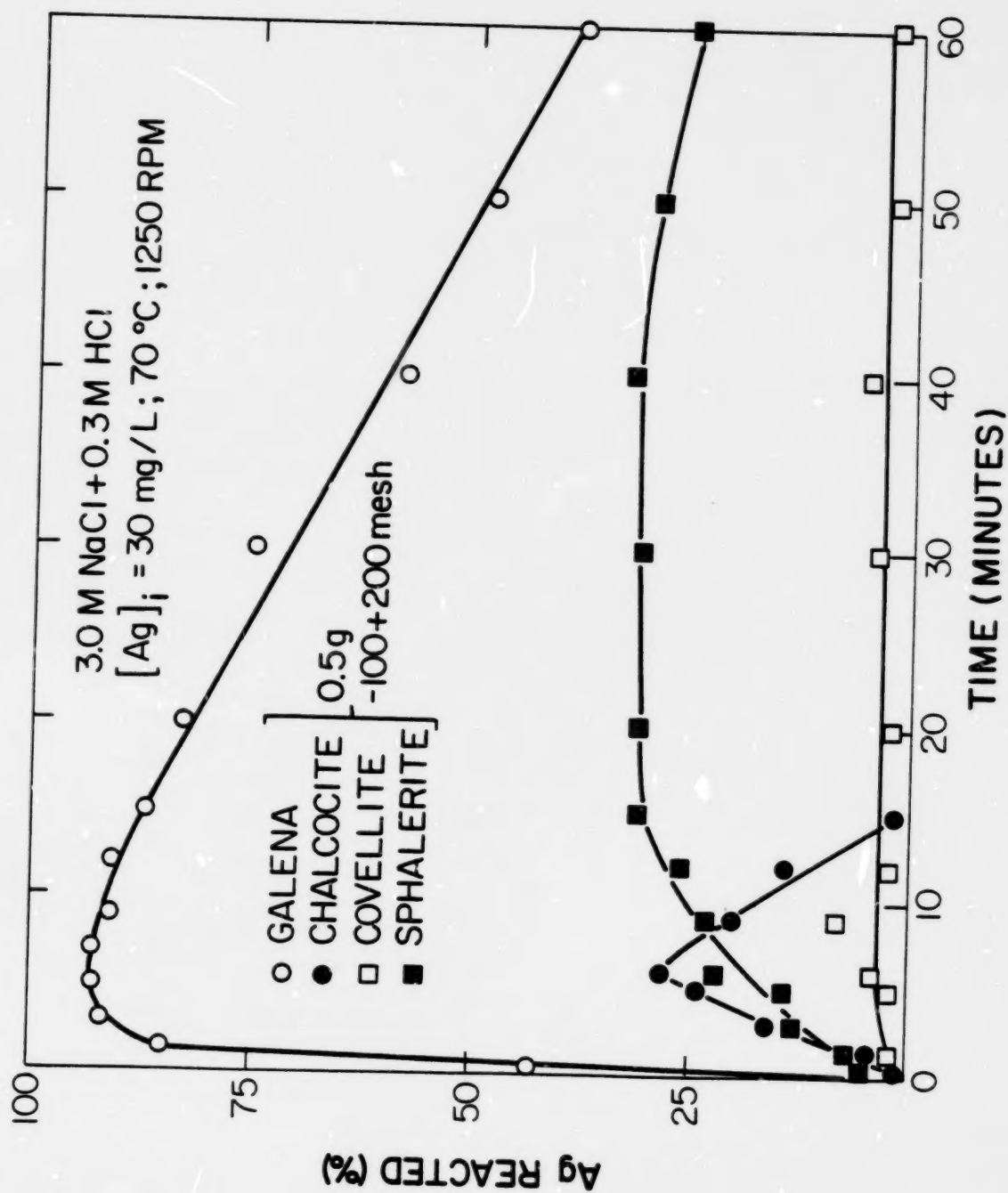
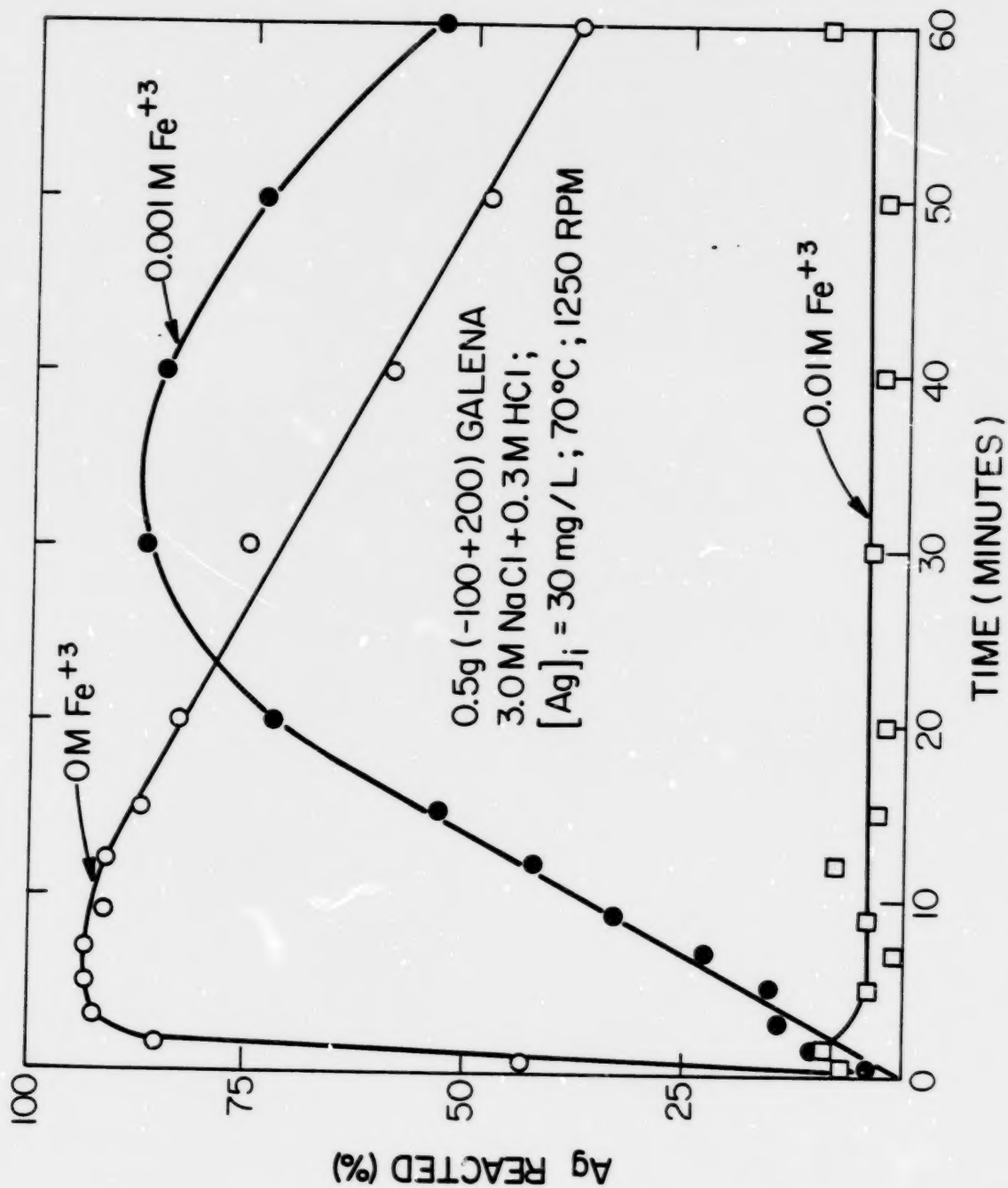


FIG 9



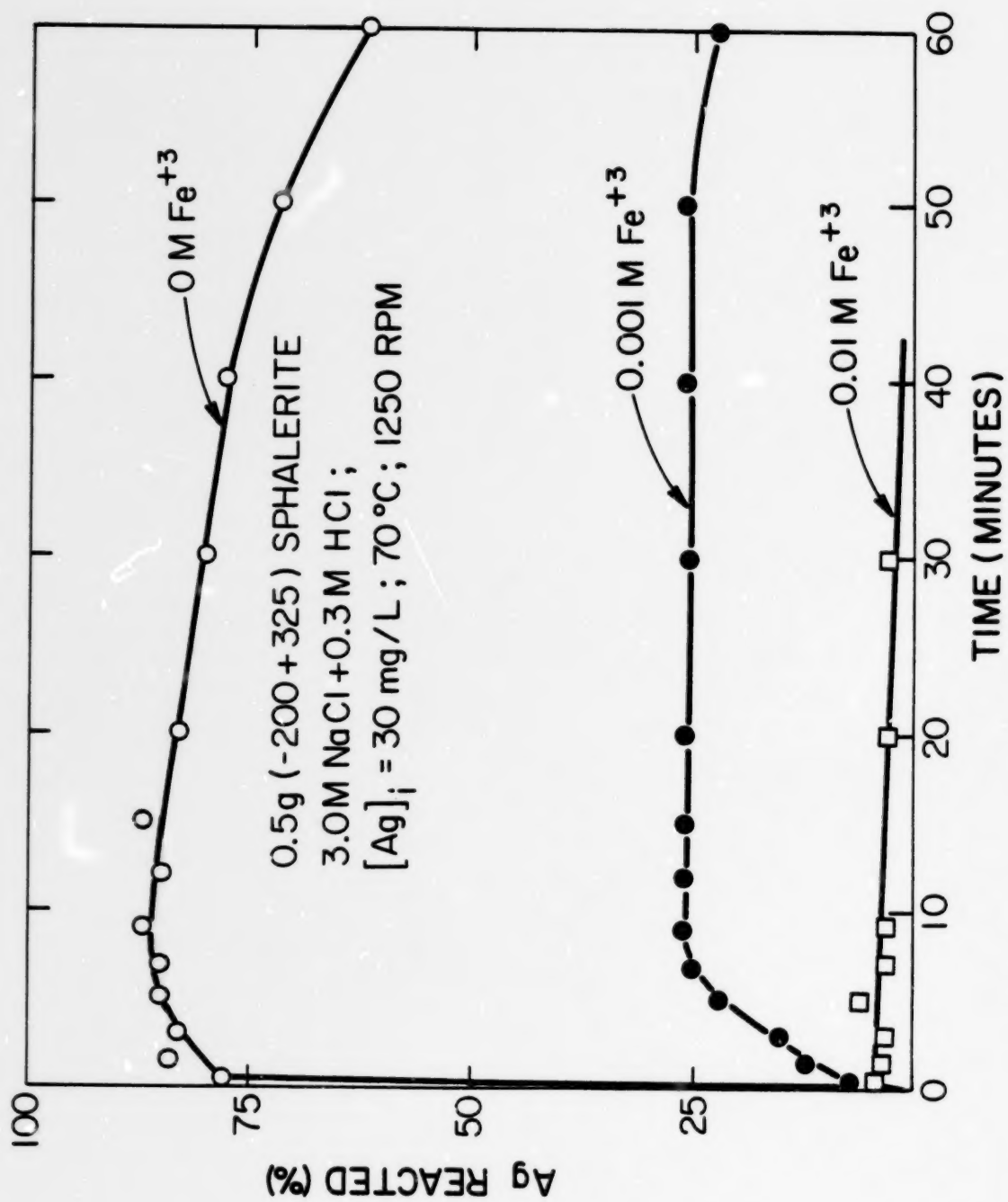


FIG. 11